



# ***In situ* Measurement of the Infrared Spectral Extinction for Various Chemical, Biological, and Background Aerosols**

**by Kristan P. Gurton, David Ligon, and Rachid Dahmani**

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**Computational and Information Sciences Directorate, ARL**

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14. ABSTRACT <p>We conducted a series spectral extinction measurements on a variety of aerosolized chemical and biological simulants over the spectral range 3–13 <math>\mu\text{m}</math> using conventional Fourier transform infrared (FTIR) aerosol spectroscopy. Samples consist of both aerosolized particulates and atomized liquids. Materials considered include <i>Bacillus subtilis</i> endospores (BG), lyophilized ovalbumin, polyethylene glycol (PEG), dimethicone (SF-96 grade 50), and three common background materials (Kaolin clay [hydrated aluminum silicate], Arizona road dust [primarily <math>\text{SiO}_2</math>], and diesel soot). Aerosol size distributions and mass density were measured simultaneously with the FTIR spectra. As a result all optical parameters presented here are mass-normalized, i.e., (<math>\text{m}^2/\text{g}</math>).</p> <p>In an effort to establish the utility of using Mie theory to predict such parameters, a series of calculations were conducted. For materials in which the complex indices of refraction are known, e.g., silicone oil (SF-96 grade 50) and Kaolin, measured size distributions were convolved with Mie theory and the resultant spectral extinction calculated. Where there was good agreement between measured and calculated extinction spectra, absorption, total scattering, and backscatter were also calculated.</p>					
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We would like to thank the Edgewood Chemical and Biological Center for their continued support and sponsorship in the area of aerosol research. In particular, we are extremely appreciative to Dr. Richard Vanderbeek and Dr. Alan Samuels for their technical expertise and the insightful conversations that aided us in the work.



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## 1. Introduction

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Currently, there is a great effort underway within both the defense and academic research community to develop an optical-based technique that is capable of detecting the presence of harmful airborne biological or chemical agents. In order to accomplish this, researchers require a set of fundamental parameters that describe the electromagnetic (EM) interaction with micron-sized particles (i.e., extinction, absorption, total-scatter, and backscatter coefficients). This report is an extension of prior work conducted as a “basic parameters” study designed to provide optical cross-sections for a variety of aerosol materials used to simulate the presence of more harmful agents (1). For many common naturally occurring aerosols, such parameters are readily available (2,3). However, for materials that are derived from biological and/or chemical matter, measured *in situ* optical parameters in the infrared (IR) are woefully lacking.

In addition, for those who wish to calculate optical cross-sections, a similar knowledge of the bulk complex indices of refraction and particle size distributions is required. Often Mie theory (only truly appropriate for perfect spheres) are applied to media in which particle geometry is quite complex. Validity of these calculations are sometimes questionable particularly when the aerosol particle dimensions are comparable to the wavelength in question. Similarly, issues arise with experimental techniques used to measure the complex indices of refraction for particulate material (4). The preferred method for deriving indices in the IR involve compressing the materials into small quasi-transparent pellets. Transmission and near-normal reflectance measurements are conducted on these pellets over a given spectral range. A Kramers-Kronig relation is then applied to the reflection spectra and resultant complex indices are calculated. However, large uncertainties arise in spectral regions where anomalous dispersion for the material is great (i.e., regions where the absorption is strong). It is often these regions of strong absorption that are of greatest interest to the researcher. As a result, we attempt to address some of these issues of accuracy for certain calculated parameters in the IR, when the aerosols are not necessarily spherical, and whose indices of refraction are in question. In practice, accurate knowledge of both size distribution and complex indices of refraction are usually very difficult to acquire. In a report soon to follow we examine to what degree such unknown quantities can be “inverted,” using only spectral extinction and/or backscatter (5).

The materials considered here may be broken into three distinctly different groups. The first group is characterized as typical biological simulants, which include *Bacillus subtilis* var. *niger* (BG) endospores (a common soil-based bacteria) and dry powdered ovalbumin (lyophilized egg white).

The second group we considered consisted of two common chemical species, polydimethylsiloxane (SF-96 grade 50) and polyethylene glycol (PEG 200), and represented our chemical simulants. These two chemicals were chosen due to their relatively low vapor pressure, which allowed good droplet formation with little or no evaporation.

The final group consisted of several “background” aerosols often present in the atmosphere (i.e., Kaolin clay [hydrated aluminum silicate], Arizona road dust [primarily SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>] and diesel soot).

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## 2. Experiment

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All samples presented here were provided by the Edgewood Research Development and Engineering Center (ERDEC), Aberdeen Proving Ground, MD. However, much of these materials are quite common and can be acquired through conventional chemical suppliers.

Aerosol samples derived from solid material (e.g., BG, Kaolin, and ovalbumin), were milled to produce particle distribution within an approximate size range of  $0.7\text{ }\mu\text{m} < \text{diameter} < 10\text{ }\mu\text{m}$ . These powdered materials were effectively aerosolized by injecting pressurized dry air through a cylindrical nozzle that contained a spiraling array of fine stainless-steel wires. A vortex created within the nozzle effectively separated and dispersed the dry particles with minimal agglomeration. Care was taken to properly adjust the air pressure to levels that resulted in good particle separation while remaining low enough to avoid fragmentation of the particles below their milled diameters.

Liquid samples were dispersed using a pharmaceutical nebulizer. These nebulizers are specifically designed to produce particle distributions within the respiratory range of  $0.8\text{ }\mu\text{m} < \text{diameter} < 4.0\text{ }\mu\text{m}$  when pressurized to 10 psi.

We have found that depending on the viscosity of the liquid, generated size distributions can be shifted to smaller diameters by slightly increasing the pump pressure. As a result both PEG 200 and SF-96 grade 50 were atomized using a pressure of 20 psi, which produced a distribution with a modal diameter  $0.82\text{ }\mu\text{m}$ . Particle-size distributions were measured *in situ* using a commercial particle-size spectrometer, TSI Aerodynamic Particle Sizer Model 3321. Resultant size distributions for aerosolized particulates and liquid droplets are shown in Figure 1. Because TSI instrument use a “time-of-flight” method to predict an aerodynamic diameter, accurate values for the “bulk” density of the aerosol particle is required. Unfortunately, for aerosols that were highly amorphous or that contain many different constituents, accurate bulk densities were unattainable (e.g., soot and Arizona road dust). As a result, size distributions for those materials are not presented. However, antidotal evidence-based aerosol settling rates and video-microscopy images of particles captured on slides suggest distributions similar to what is seen for Kaolin (i.e., a large submicron component with a modal diameter between 1 and  $5\text{ }\mu\text{m}$ ).

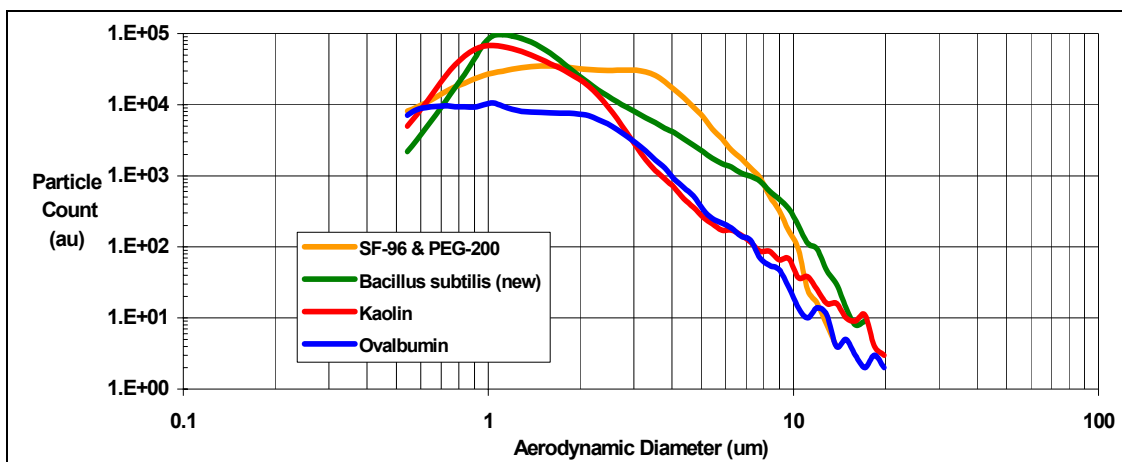


Figure 1. Measured size distribution for particulates and liquid droplets.

The primary transmission measurements were conducted in a 0.8-m<sup>3</sup> aerosol chamber which provided an optical path length of 0.61 m. Dispersed aerosols were gently drawn into the chamber using a small-area recirculating fan. Continuous monitoring of humidity levels was conducted using a filtered dew-point hygrometer that was inserted through the walls of the chamber. Because dry air was used to flush the various optical windows, water vapor levels were kept to a minimum. Relative humidity levels for most runs were typically < 8%, which is representative of a fairly dry environment.

We obtained IR transmission spectra using a high-resolution (0.02 wave number) Bomem DA2.02 Fourier transform IR (FTIR) spectrometer. For this study, the spectrometer was operated in a transmission mode (i.e., spectral attenuation was measured by placing the aerosol chamber between the source and the interferometer). A broadband IR Nernst glower was collimated with a ZnSe condensing lens assembly and projected through the aerosol chamber with two BaF<sub>2</sub> transmission windows that were fitted with dry-air flushes. Transmitted light was coupled to the interferometer with a gold-surfaced f/4 off-axis parabola. Because spectra derived from aerosols are devoid of any fine structure often seen in vapor spectra, the FTIR was operated at a spectral resolution of 4.0 cm<sup>-1</sup>.

Aerosol mass densities (g/m<sup>3</sup>) were periodically measured during the run using conventional dosimetric techniques (6). Aerosol mass samples were collected by drawing known volumes of the air-particle mixture onto polycarbonate filters, nominal pore-size 0.20 μm, for predetermined periods of time. Results from the dosimetric sampling were then used to mass normalize the measured spectral extinction, (m<sup>2</sup>/g).

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### 3. Results

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Interferograms were recorded before, during, and after each aerosol dispersion. A Bartlett apodization was applied to each interferogram before the background spectra were removed. Transmission was converted to extinction using a Beer's law relation. Results were mass normalized by dividing the path-integrated extinction,  $(1/m)$ , by the corresponding aerosol density,  $(g/m^3)$ .

For materials in which complex indices of refraction were available from prior studies (Figures 2–4), Mie theory calculations were conducted using the appropriate size distribution shown in Figure 1 (7). Where agreement between the measured and calculated extinction was reasonable, the calculated spectral absorption, total scatter, and backscatter are also presented (Figures 5–7). For ovalbumin, PEG 200, diesel soot, and Arizona road dust in which no such indices were available, only the measured spectral extinction is presented (Figure 8).

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### 4. Comments

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When comparing measured extinction with Mie calculated values (Figures 5–7) one should keep in mind that all the complex indices used in the calculations were gathered from prior studies and may not accurately represent the materials used here. Nevertheless, we thought it informative to show such comparisons because it is often the case that one must rely on previously published constants to compute various optical parameters.

For the three materials presented with refractive indices (i.e., BG, Kaolin, and SF-96 grade 50), the refractive index for SF-96 grade 50 is least likely to vary from one measurement to the next because it is a highly standardized chemical where production is tightly monitored. In addition, because it is a liquid, we know with certainty that the nebulized particles must be spherical and thus most appropriate for applying Mie theory. As one can see in Figure 6, agreement between measured and calculated spectral extinction is quite reasonable. Agreement for the location of the peaks and valleys for the spectral extinction is quite good. However, agreement between the relative magnitude between the two is not as good and is attributed to uncertainties in the size distribution (Figure 1). In general, the underpredicted values for the Mie extinction is synonymous with a slightly broader size distribution than seen in Figure 1. Nevertheless, where agreement is acceptable (e.g., 3–8.5  $\mu m$  and 10–12  $\mu m$ ), Mie computed absorption, scatter, and backscatter should be fairly representative of the true values.

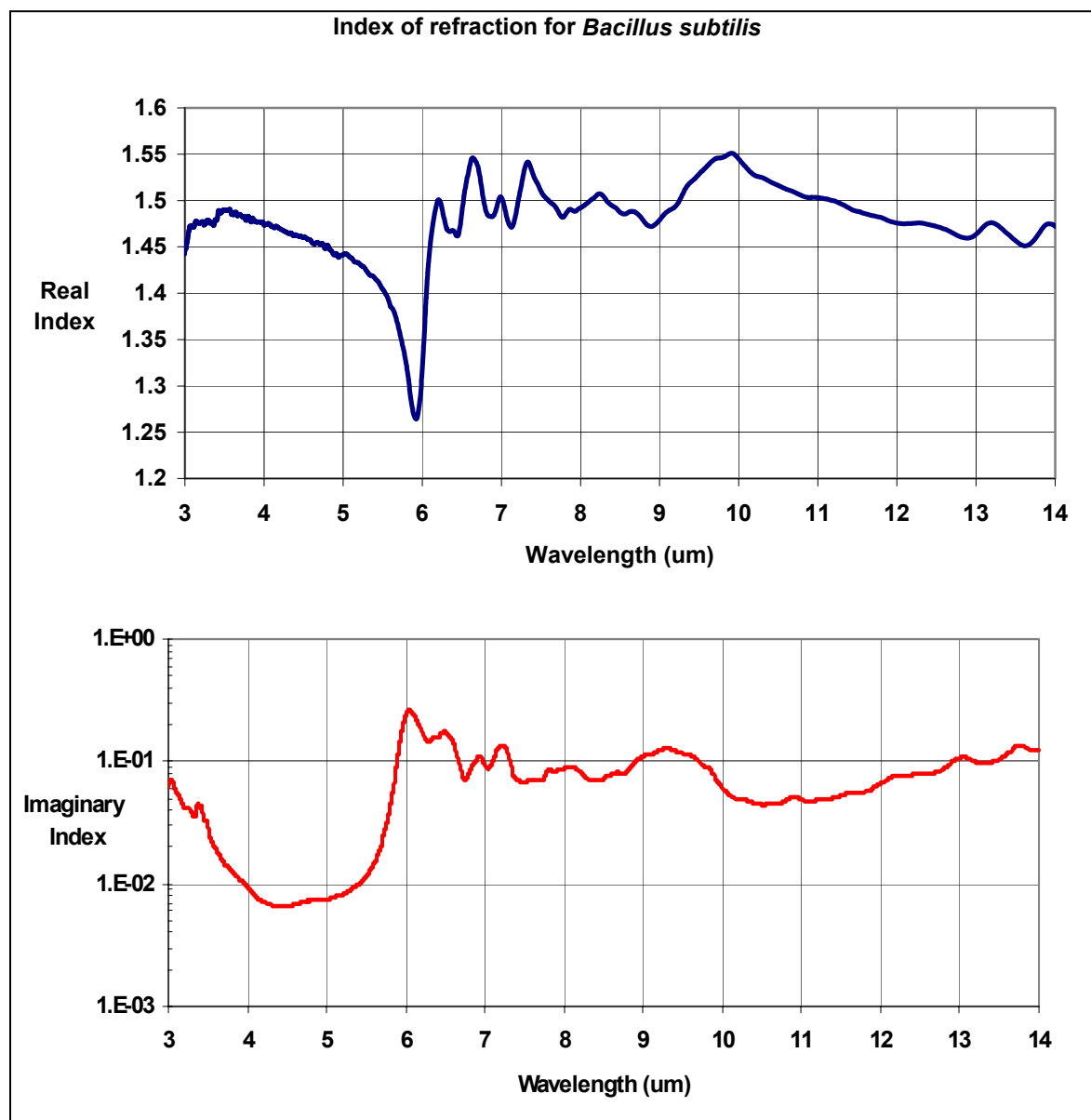


Figure 2. Complex indices of refraction for *Bacillus subtilis* endospores.

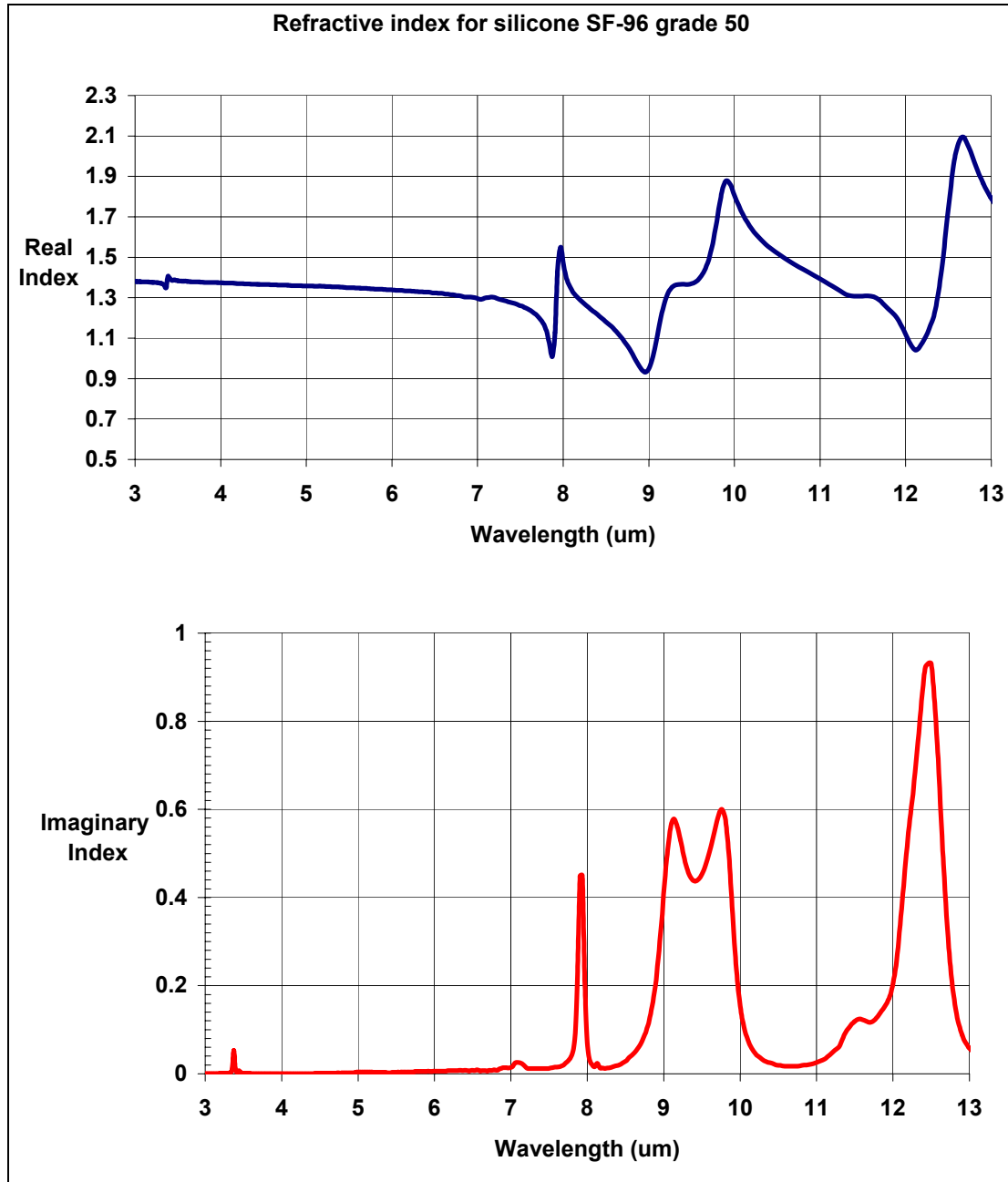


Figure 3. Complex indices for liquid silicone SF-96 grade 50.

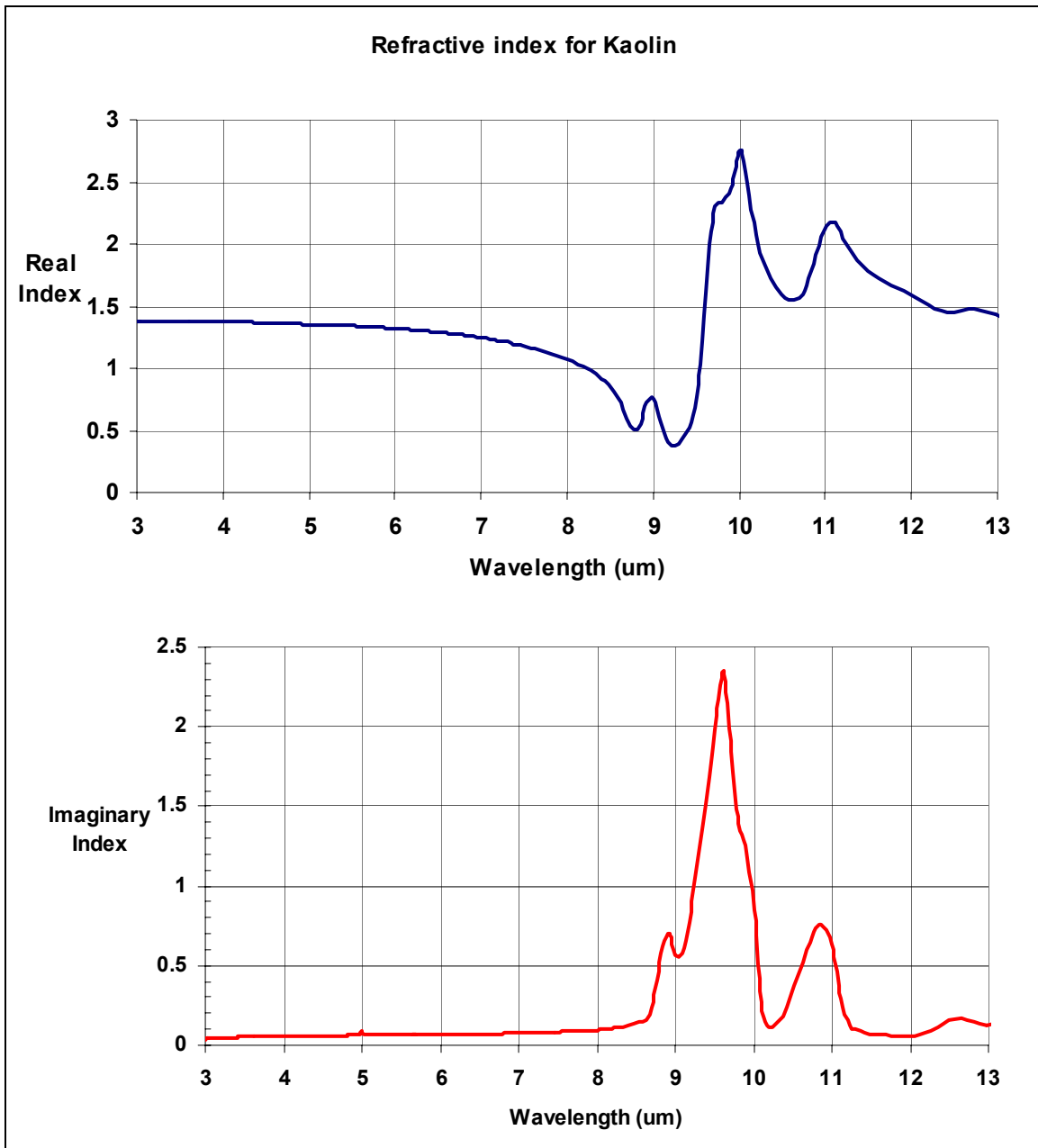


Figure 4. Complex indices of refraction for Kaolin clay.



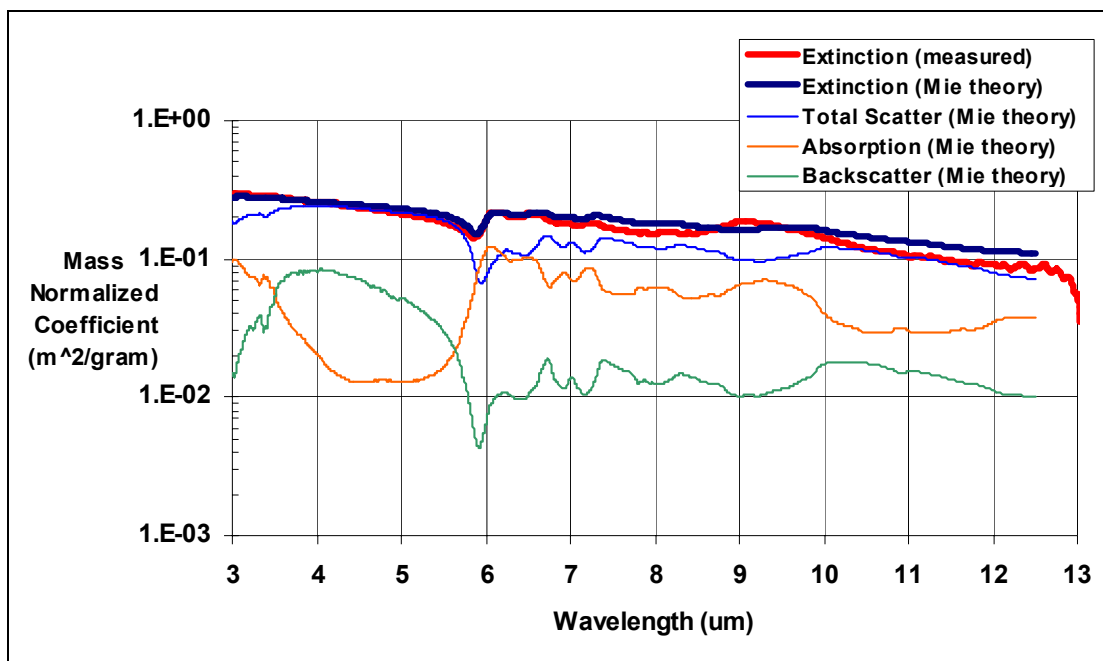


Figure 5. Measured (thick red) and calculated (thick blue) spectral extinction for aerosolized *Bacillus subtilis* endospores. Also shown are the Mie theory predicted total scatter (thin blue), absorption (orange), and backscatter (green).

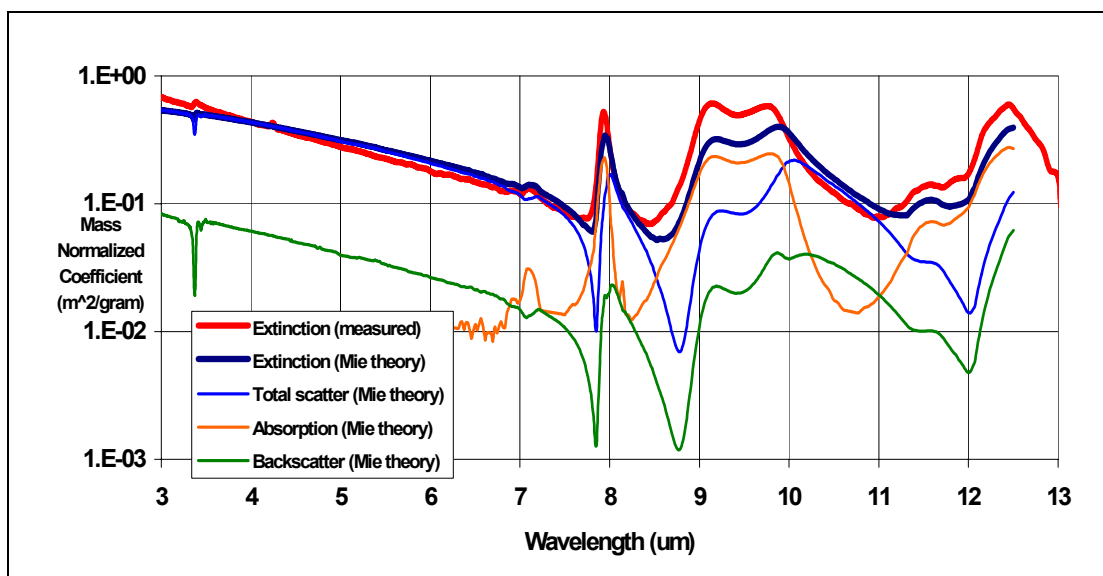


Figure 6. Measured (thick red) and calculated (thick blue) spectral extinction for nebulized liquid silicone SF-96 grade 50. Also shown are the Mie theory predicted total scatter (thin blue), absorption (orange), and backscatter (green).

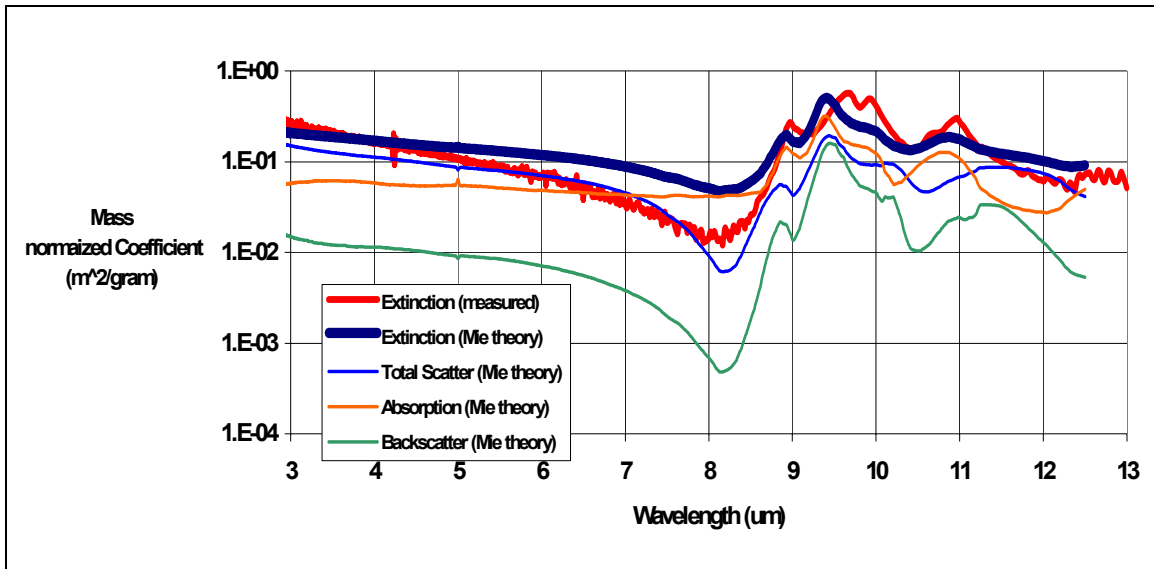


Figure 7. Measured (thick red) and calculated (thick blue) spectral extinction for aerosolized Kaolin clay. Also shown are the Mie theory predicted total scatter (thin blue), absorption (orange), and backscatter (green).

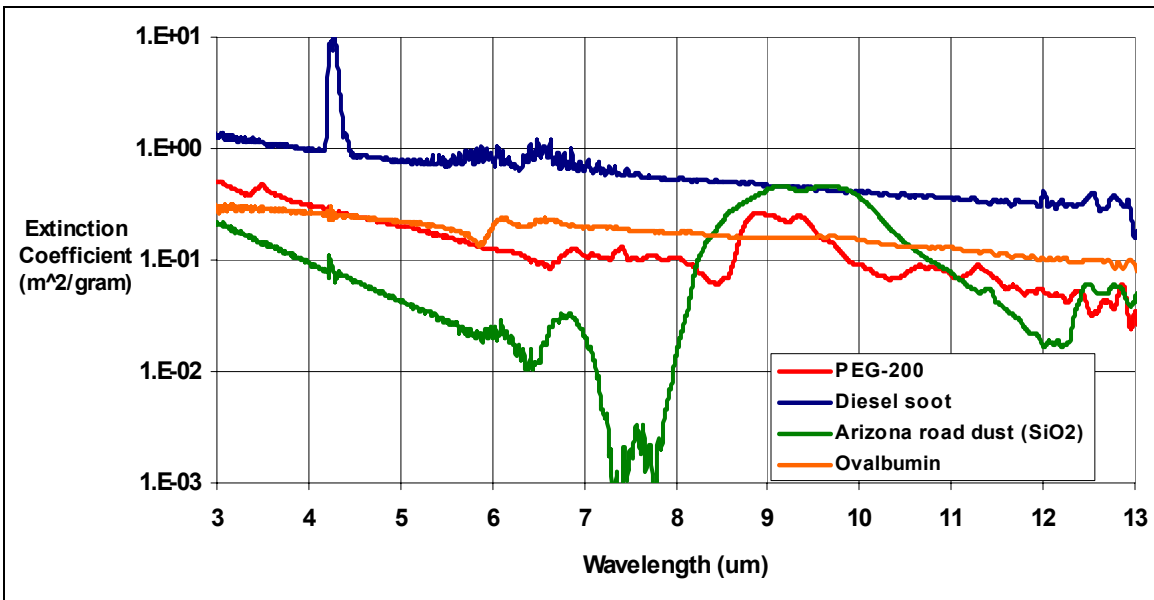


Figure 8. Measured spectral extinction for PEG 200, diesel soot, Arizona road dust, and ovalbumin.

As for the *Bacillus subtilis* (BG) samples, little is known about the exact form and conditions in which the indices were measured by Query (7). What is certain is that the BG sample(s) provided by ERDEC was grown, cleaned, and milled from a completely different batch than used by Query nearly a decade prior. In lieu of this and given the fact that growth media used for producing bacterial endospores can be quite variable, agreement seen in Figure 5 is good. One notable region of divergence occurs in the long wave IR between 9 and 10  $\mu\text{m}$ . Although difficult to see in Figure 5, the measured spectral extinction shows a modest peak at 9.1  $\mu\text{m}$ , but the calculated spectra using Query's indices show this peak quite suppressed and shifted toward 10  $\mu\text{m}$ . Although researchers have attributed this peak to absorption by phosphates and/or polysaccharides, we believe a portion of the increased extinction seen in the measured spectra between 9 and 10  $\mu\text{m}$  may be due to a residual silicate used during the milling process.

Perhaps the least certain complex index used was that of Kaolin. It is well known that the optical properties for Kaolin are quite variable, depending upon the geographic location where it was mined (i.e., Kaolin is routinely sourced from North America, China, and Europe). As a result, good agreement between measured and calculated extinction was not expected. As seen in Figure 7, the spectral features of the Mie calculated extinction appears suppressed when compared to the measured values.

Figure 8 shows the measured mass-normalized spectral extinction for PEG 200, Arizona road dust, ovalbumin, and diesel soot. As mentioned before, complex indices of refraction were not available for these materials and as a result no Mie calculations are presented. However, we will note several key aspects for the various spectra seen in Figure 8.

First, the two noticeable features seen in the extinction spectra for diesel soot seen at 4.25  $\mu\text{m}$  and the fine structure seen between 5 and 8  $\mu\text{m}$  are due to gaseous absorption of  $\text{CO}_2$  and water, respectively, that were generated during the burning of diesel fuel and proved extremely difficult to remove using conventional FTIR background subtraction methods. Prior studies have shown this spectra to be devoid of any real structure and decreases monotonically with increasing wavelength (8).

Although the preparation/dissemination of the PEG 200 liquid was relatively straightforward, we did notice during several runs the emergence of strong water vapor absorption lines when the liquid samples were left in the open for periods of several hours due to the fact that PEG 200 is extremely hygroscopic. For this reason, care should be taken when using PEG 200 chemical simulant for aerosol research because water uptake for atomized samples is very likely except in the most arid of environments.

The extinction spectrum for ovalbumin turned out to be somewhat surprising. As one can see by comparing ovalbumin spectra with the Mie calculated extinction spectra for BG seen in Figure 5, the two spectra are nearly identical (Figure 9).

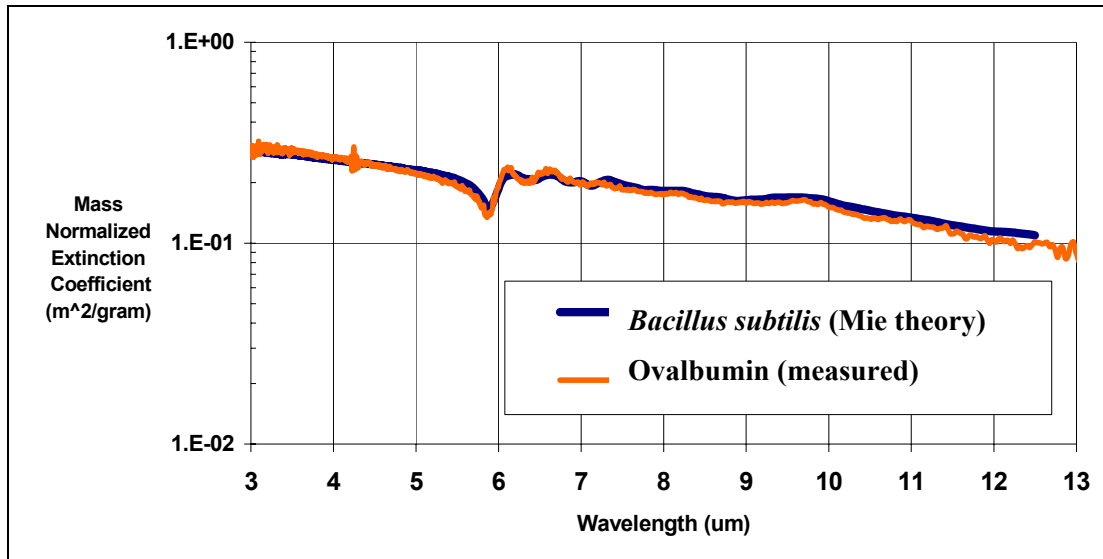


Figure 9. Comparison of the measured extinction spectra for ovalbumin (lyophilized egg white) with a Mie calculation using the indices of refraction for *Bacillus subtilis* endospores (BG).

Although not definitive, based on this comparison, it seems that one might be able to use with this comparison the expected complex indices of refraction for ovalbumin should be similar to that of BG seen in Figure 2. Where cost and the availability of a good biological simulant are an issue, using ovalbumin powder may suffice. This would be particularly useful for field tests in which large quantities of a simulant are required.

The last material shown in Figure 8 is that of a somewhat standardized background aerosol called Arizona road dust obtained from Powder Technology Inc., Burnsville, MN. Because Arizona road dust is well characterized it has become a popular natural obscurant used in many radiative transfer measurements. The particular type used for this study was sized to have a nominal diameter of  $1.13 \mu\text{m}$  with a standard deviation  $0.385 \mu\text{m}$ . Chemical analysis shows the following compounds as a percentage of mass: 76%  $\text{SiO}_2$ , 15%  $\text{Al}_2\text{O}_3$ , 4%  $\text{Fe}_2\text{O}_3$ , 4%  $\text{Na}_2\text{O}$ , as well as traces of  $\text{CaO}$ ,  $\text{MgO}$ , and  $\text{TiO}_2$ .

Finally, as part of this study we were provided with an unusual material called Cab-O-sil. Cab-O-sil is a synthetic, amorphous, untreated fumed silicon dioxide that is composed of fine sub-micron  $\text{SiO}_2$  particles. Used commercially as a thickening agent for food and cosmetics, this silica “aerogel” is extremely amorphous (94% of its volume is air) and is sometimes used as a fluidizer to improve aerosol dissemination efficiencies. As a result, we were asked to produce spectra for this possible residual compound. However, difficulties with extreme agglomeration arose during the FTIR measurement. Various attempts to disperse dry well-separated Cab-O-sil particles failed. Resultant measured spectra showed little wavelength dependence regardless of the concentration (Figure 10). These “flat” spectra are synonymous with the presences of very

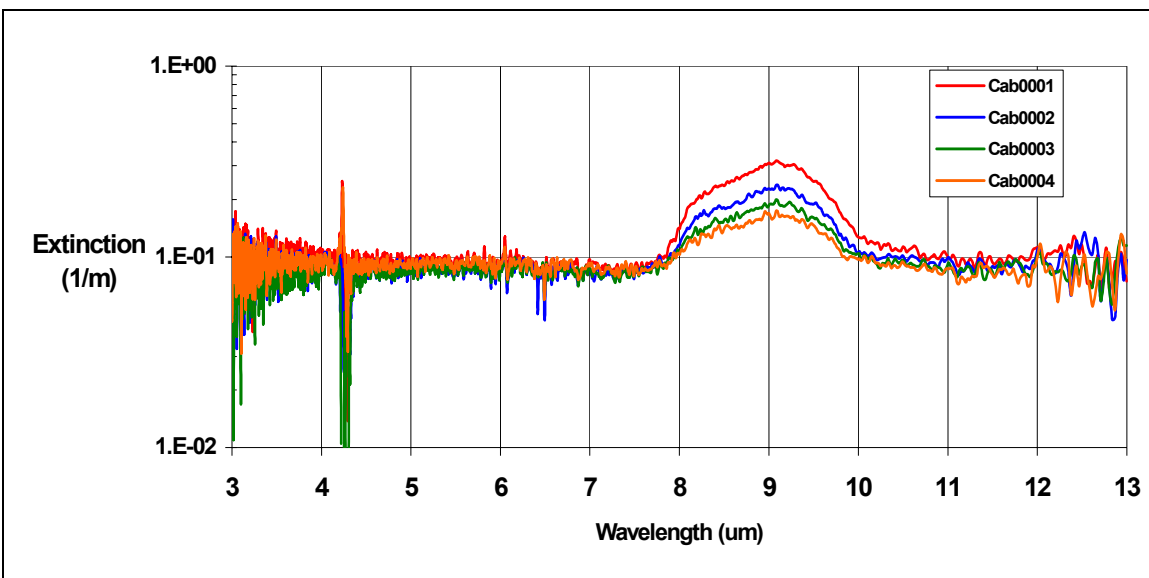


Figure 10. Measured extinction for agglomerated Cab-O-sil aerogel at various concentrations. The flat spectral extinction is synonymous with very large particles (compared with the wavelength).

large particles when compared to the wavelength (i.e., the extinction mechanism is primarily due to the particle's geometric cross-section rather than any diffraction effects). Efforts to measure the *in situ* aerosols density also failed because we were unable to collect enough measurable mass on the dosimetric filter substrates during the spectral runs. Nevertheless, one can see the obvious absorption between 8 and 10  $\mu\text{m}$  due to  $\text{SiO}_2$ , which is also seen in the Arizona road dust spectra.

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